# MECHANICAL AND PHYSICOCHEMICAL PROPERTIES TESTS IN COMPOSITE RESINS: INDICATIONS AND APPLICABILITY

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## ABSTRACT

The relationship between the mechanical properties of dental materials and their intraoral performance has been the subject of extensive studies, which have resulted in the identification of significant correlations. Physicochemical properties refer to the characteristics that influence the interaction of materials with the oral environment, encompassing factors such as reactivity, durability, and stability. Among the main physicochemical properties, chemical composition, solubility, water absorption, biocompatibility, color and aesthetics stand out. This chapter addresses the main dental tests to evaluate mechanical and physicochemical properties of resin materials, based on a literature review carried out in the PubMed and SciELO databases, highlighting their characteristics and indications. Flexural strength testing can be related to the clinical problem of deformation, and its data provide indications of the strength properties and modulus of elasticity of a dental material. The hardness of a material corresponds to its resistance to local deformation, not being an intrinsic property of the material, it is the result of a measurement procedure. Sorption and solubility testing is crucial, as water sorption can negatively affect the dimensional stability and mechanical properties of the material,

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impacting its long-term durability. Materials that absorb more water can show expansion and loss of strength, compromising their clinical performance. Minimum values expected by the ISO standardization of these tests are requirements in the development of more durable and reliable materials.

**Keywords:** Mechanical Tests. Solubility. Dental Materials. Composite Resins.

## INTRODUCTION

The mechanical properties of dental materials are fundamental for the evaluation of the behavior and resistance of these materials in response to applied forces or stresses, in order to characterize their ability to resist permanent deformation or fracture under the conditions found in the oral cavity. These properties include the analysis of the acting forces, motion, deformation, and stresses that materials undergo. In dentistry, the most relevant mechanical properties include: compressive strength, ductility, modulus of elasticity, flexural strength, fracture toughness, hardness, impact strength, shear strength, and tensile and torsional strength, among others. Understanding these properties is crucial for proper material selection and for ensuring the effectiveness and durability of dental restorations (Anusavice; Shen; Rawls, 2013; Reis *et al*., 2020; Lima *et al*., 2023).

The relationship between the mechanical properties of dental materials and their intraoral performance has been the subject of extensive studies, which have resulted in the identification of significant correlations. Tyas (1990) reported a substantial inverse correlation between surface chipping and mass fracture of composites, in relation to fracture toughness, modulus of elasticity and diametrical tensile compressive strength. Similarly, Ferracane *et al.* (1999) observed an analogous correlation between fracture toughness and marginal breakage of composite resins. Ferracane and Condon (1999), when analyzing microfill, minifill and midfill composites with a wear simulator, demonstrated a strong inverse correlation between marginal breakage and fracture toughness. Additionally, Ferracane *et al.* Wang et al. (1997) reported a robust inverse correlation between wear and fracture toughness, as well as between wear and flexural strength, while a weaker inverse correlation was observed between wear and hardness. In addition, fracture toughness, along with flexural strength and flexural modulus, has been proposed as predictive indicators of clinical wear of composite resin restorations. Thus, all these mechanical properties are fundamental for the characterization of the composite resin in relation to its clinical performance.

A fundamental aspect in determining the mechanical properties of dental composites is the correlation between their parameters. Although mechanical properties are often considered independent since they describe different aspects of material behavior, universal correlations can provide a more efficient and simplified approach to the characterization of material properties, allowing unknown properties to be inferred based on the identification of a single parameter. A notable example of this practice is the correlation between hardness and modulus of elasticity, which has been widely employed to estimate the modulus of elasticity of dental composite resins from hardness measurements. This relationship not only facilitates the evaluation of mechanical properties, but also contributes to the proper selection of materials in clinical applications (Li *et al.,* 2009; Thomadis *et al.,* 2013).

Physicochemical properties refer to the characteristics that influence the interaction of materials with the oral environment, encompassing factors such as reactivity, durability, and stability. Among the main physicochemical properties, chemical composition, solubility, water absorption, biocompatibility, color and aesthetics stand out. In clinical dental practice, these materials often require the combination of different components to obtain a putty or liquid that will be handled, adapted, and molded for the desired application. The flow of the material during its handling stages is called rheology. The constant temperature variations in the oral cavity make it essential to understand the thermal properties of restorative materials, since dental pulp is sensitive to exposure to thermal extremes (Reis *et al.,* 2020).

Mechanical, chemical, physical and biocompatible properties are essential. Dental materials need to withstand different mechanical forces, exposure to moisture, various temperatures, and fluctuating pH values. This leads to aging, which changes mechanical properties over time. Temperature influences aging processes through increased reaction rate, which accelerates chemical degradation (Gorning, 2022).

This chapter aimed to present the main dental tests used to evaluate the mechanical and physicochemical properties of resin materials. To this end, a literature review was carried out in the PubMed and SciELO databases, in order to identify the main characteristics of these tests and their respective indications.

## STRESS *versus* DEFORMATION

Tension can be defined as the reaction generated by the application of a load on a body, which acts in the opposite direction to the external direction (Reis, 2021). The dental structure and dental materials used in oral rehabilitation are susceptible to the action of factors that generate tensions such as: masticatory forces, parafunctional habits, functional changes and activities associated with posture and musculature. The forces generated by loads result in structural stresses, if such stresses become excessive and exceed the elastic limit, structural failure can occur, depending on the characteristic of the material (May *et al.,* 2012; Vianna *et al*., 2018).

Among the mechanical properties, there are different types of stresses that develop according to the type of load on the material. These can be classified as: simple (traction, shear and compression) or complex (bending and torsion). Tensile stress is a tension that occurs in the same line and in opposite directions, and tends to stretch and elongate the material. On the other hand, shear stress is the result of the application of a load in the opposite direction, but parallel, this can also be promoted by torsional stresses. Compressive stress is generated when a load is applied to the body in the same direction, where a point is mobile, and this is directed to the fixed point, with the aim of compressing the material (Anusavice; Shen; Rawls, 2013).

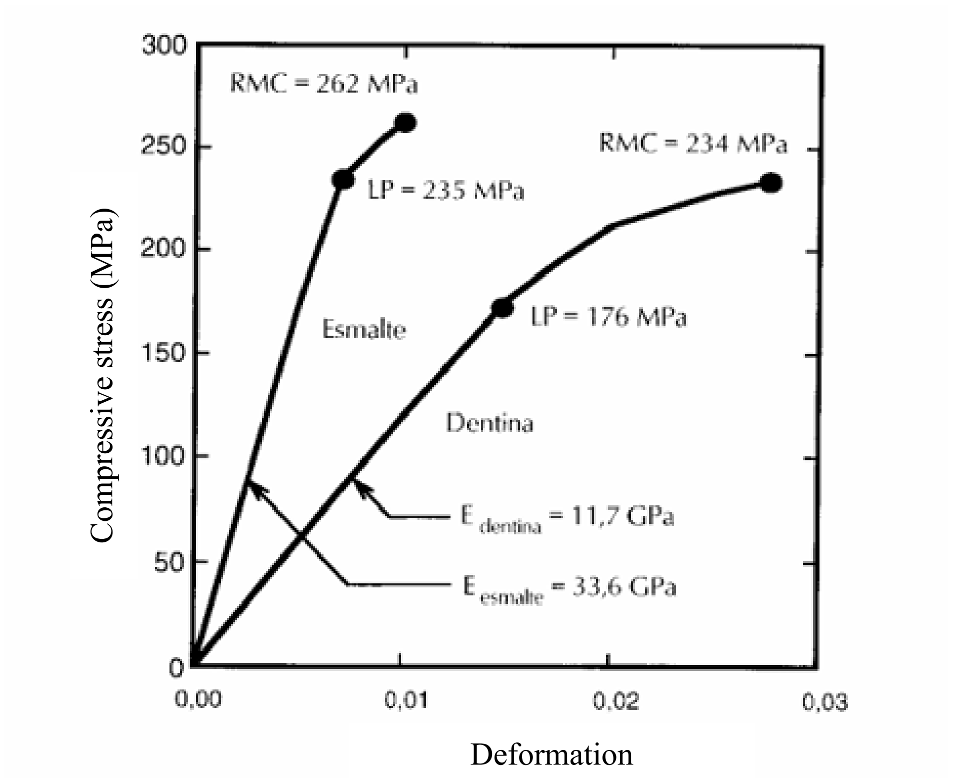
In complex stresses, there is the bending stress, which can be of three points, where the three simple stresses act on the material, being the traction in the lower zone, shear at the ends and compression in the upper zone. Finally, torsional stress occurs when the ends of the material are rotated in the opposite direction (Anusavice; Shen; Rawls, 2013).

When a stress is applied, a strain is generated in response to the applied force, and it overcomes it, causing changes in the dimensions of the material. Deformation can be classified as elastic (there is the application of a tension that causes changes in the material, however, when it is removed, the particles of the material return to their original dimensions), and plastic (there is the application of a tension that causes changes in the material, however, when it is removed, the force goes to zero, but particles of the material do not return to their original dimensions,  because they have already been permanently deformed) (Reis, 2021).

## STRESS-STRAIN CURVE

For example, the stress-strain curve of a material represents the relationship between the stress and the strain of that material under the application of a force, and can be obtained by tests carried out on the materials, to obtain information about their characteristics, and to provide safety, before launching it on the market (Goodno; Gere, 2017).

Stress-strain curve of enamel and dentin submitted to compressive strength test:



Source: Anusavice; Shen; Rawls, 2013; Stantord, 1960. Legend: The values of the proportionality limit (LP); maximum compressive strength (RMC) and modulus of elasticity (E) are shown in the curve.

The vertical line represents the stress exerted on the material and the horizontal line the deformation caused in response to the applied stress. The linear curve (from points 0 to LP, i.e., the linear curve within the elastic strain region) has a significantly straighter slope than the second curve, where there is no linearity (from points LP to RMC, i.e., the curve within the plastic strain region). There are some regions and points associated with this stress-strain curve that have important mechanical significance (Anusavice; Shen; Rawls, 2013; Lin; Kang, 2021).

These will be described below:

A. **Amplitude of the Elastic Region:** This region comprises from the point of origin (0) to the elastic limit, where the elastic deformation occurs, if the force is removed, the stress goes to zero, and the particles of the material organize themselves and return to their original dimensions. In this region, according to Hooke's law, the stress must be proportional to the strain. No permanent deformation will occur, and the material will be intact with the removal of the force.

B. **Plastic Region Amplitude:** This region corresponds to the point above the proportionality limit. If the material is subjected to the action of a force above the elastic region, plastic deformation will occur. Therefore, with the removal of the force, the stress goes to zero, but the particles of the material do not return to their original dimensions, as they have already been permanently deformed. The stress will not be proportional to the strain based on Hooke's law.

C. **Limit of Proportionality:** This is indicated on the curve by the acronym LP, and can be defined as the point above which the curve ceases to be a straight line. This marks the transition between elastic and plastic deformation. It represents the maximum stress above which the stress is no longer proportional to the strain. Some authors may define proportionality limit as being the same as elastic limit, because they are close to each other, however, based on the referenced content, they will be treated as distinct.

D. **Elastic Limit:** This is the maximum stress supported by the material, so that it can return to its original dimensions with the removal of force, without suffering plastic deformation.

E. **Yield Strength (Proof Stress):** Corresponds to a small amount of plastic strain (about 0.1% to 0.2%) induced by stress and is located above the yield strength on the stress-strain curve.

F. **Modulus of Elasticity:** This is the relative stiffness of a material, it is measured in the elastic region of the stress-strain curve. This is constant, and is not altered by the elastic or plastic tension generated on the material.

G. **Maximum Resistance:** In the graph, whose resistance test is the compression test, this is given by the acronym RMC. In materials mechanics, failure is defined as the moment when the specimen fractures completely into more than one fragment. Failure resistance is the maximum stress that the specimen can withstand before failure, which can occur in the elastic or plastic region, depending on the mechanical characteristic of the material.

## FLEXURAL STRENGTH

Strength is not an inherent property of composite resin, i.e., it is not completely predictable solely based on the chemical composition or internal structure of the material, it may depend on the clinical context and external variables. In this sense, to analyze the bending of a material, the force is applied in different ways that create internal stresses within the material, and it is then measured and recorded as fracture strength. Flexural strength testing can be related to the clinical problem of deformation, and its data provide indications of the strength and modulus of elasticity properties of a dental material (Ilie *et al.,* 2017).

It is possible to state that the strength of a material involves its composition and quality of preparation (internal porosity and surface failures), and it is therefore necessary to ensure the preparation of totally solid specimens without significant failures. These specifications are indicated by ISO 4049/2019 - *Dentistry-Polymer-Based Restorative Materials* , which guides tests on dental composites.

The flexural strength test develops tensile, compressive and shear stresses and according to the literature, there is a correlation between flexural strength and clinical fractures of composite resin restorations, which can predict the clinical wear of the composite. Cycling that simulates aging of the material can improve this relationship between laboratory data and clinical studies (Ferracane, 2013).

To evaluate the strength of dental composites, the flexion test can be performed in three ways, namely: three-point flexion (A), four-point flexion (B) and biaxial flexion (C) (Ilie *et al.,* 2013).

A. **3-point bending:** the specimen will be beam-shaped with specific dimensions (approximately 10% additional material, in addition to the supports at each end), supported on two separate rollers with a specified distance, and at the top center of the beam a force will be carried to failure, and it is expected that there will be minimal plastic deformation of the specimen;
B. **4-point flexion:** the specimen should also follow rectangular conformity (beam), and should maintain lower support at two separate points so that there is an additional 10% of the specimen at each end. Regarding the upper force, two upper points apply the load symmetrically between the lower supports, creating a distributed load area. This allows you to evaluate a larger sample volume, making the test less sensitive to localized defects;
C. **Biaxial Flexion:** In this case, the specimen is disc-shaped and is supported on three points or on a ring that supports the entire circumference, providing a uniform distribution of tensile stress within the specimen. It is generally indicated for materials that are more fragile than resin composites, however, its data can be correlated with those obtained in the 3-point bending.

Composite resin-based materials are constantly introduced into the clinical reality, requiring the physical-mechanical evaluation of these materials constantly. They must be subjected to flexion forces to indicate clinical longevity when used in anterior and posterior tooth restorations (Yap *et al.,* 2018).

## MICRODUCTIBILITY KNOOP AND VICKERS

The hardness of a material corresponds to its resistance to local deformation, and is not an intrinsic property of the material, it is the result of a measurement procedure (Tabor, 1970). The determined modulus of indentation is classified in importance parallel to the modulus of elasticity determined in a flexural test, as it can allow a quick and accurate assessment of the material's resistance to deformation. In general, the hardness test uses an indenter of specific shape for each method, which will be pressed on the surface of the specimen for a certain time, obtaining the measurement of the depth of the indentation (Fischer-Cripps *et al*., 2004; Cramer *et al*., 2011).

The hardness test enables the quantitative analysis of the resistance to deformation, being calculated as the maximum applied load divided by the projected contact area. The possible methods for performing the test are: Brinell, Knoop (B), Rockwell and Vickers (A). These correspond to loading a hard indenter against the material. For these classic hardness measurements, only the plastic part of the indentation process is considered.

*ATTENTION!* →

Hardness measurement can be defined as macro, micro or nanoscale according to the applied forces and the displacements obtained (Ohki et al., 2012): macro range: 2N ≤ F ≤ 30kN; Micro range: 2 N > F; h > 0.2 µm; and nano range: h ≤ 0.2 µm (where F = Force and h = indentation depth) (ISO, 2005; ISO 4545–1, 2017).

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A. **Vickers microhardness** (ISO/CD6507-1): indent specimen with a diamond indenter, in the shape of a pyramid with a square base and an angle of 136° between opposite faces. Full charge is usually applied between 10 to 15 seconds. The two diagonals of the indentation, which remain on the surface of the material after the load has been removed, are measured using a microscope and averaged out. The test procedure can be factors influencing the operator in the optical reading of the indenter diagonals. Modern devices allow for automatic measurement of indentations.

B. **Knoop microhardness** (ISO 4545-1): the force is applied with a pyramid-shaped diamond indenter (with angles of 172.5° and 130° between the opposite edges at the vertex) for a dwell time that should not exceed 10 seconds, and the test force is maintained for 10 to 15 seconds. The Knoop hardness value is proportional to the test force divided by the projected area of the indentation. Compared to a Vickers indenter, the indenter used in a Knoop test has a more elongated shape. The Knoop method is commonly used when the indentations are very close to the edge of the sample.

The specimens can be made in disc format for microhardness tests. With the use of a stainless steel die in the following dimensions: 2 mm thick by 5 mm diameter, the material must be inserted with a spatula, therefore, a strip will need to be applied so that the mold is pressed with a glass plate to remove the excess material, and allow the leveling of the surface,  and this is kept homogeneous (Cruz; Águila, 2024).  Specimen preparation requires a low-roughness, ideally smooth, polished, and uniform surface (the maximum deviation in parallelism should not exceed 0.1 to 50 millimeters). The surface of the specimen must be completely free of any lubricants, polishing pastes, waterproof pen marks, or any visible surface flaws.

## SORPTION AND SOLUBILITY

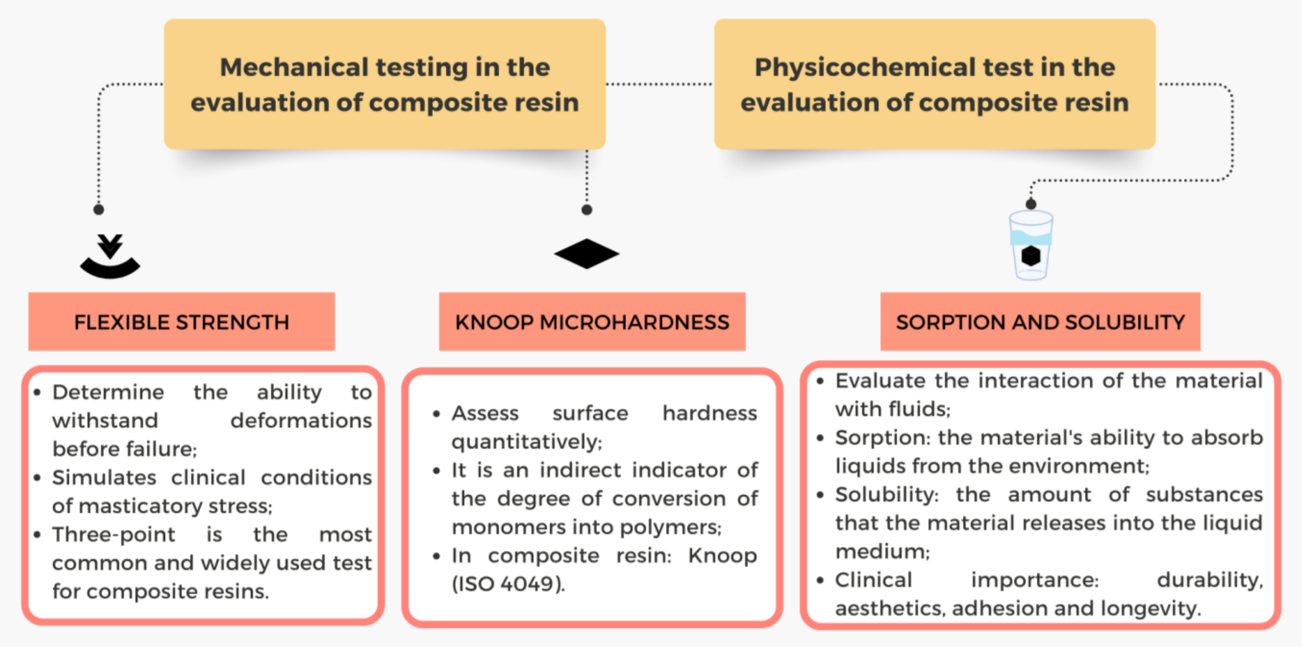
The amount of water that a resin composite can absorb is directly related to the hydrophilia of the monomers that make up the polymer matrix (Toledano *et al.,* 2003). This test is crucial, as water sorption can negatively affect the dimensional stability and mechanical properties of the material, impacting its long-term durability. Materials that absorb more water can show expansion and loss of strength, compromising their clinical performance.

Regarding solubility, this test is also essential to evaluate the behavior of dental materials, as solubility is directly related to the amount of residual monomers that can be leached into the oral cavity. Solubility is influenced by several factors, including immersion time and the characteristics of the storage medium. When test factors are controlled, other elements such as the monomeric composition, the filler used, the silane treatment, and the degree of cross-linking of the polymer also play an important role (Ortengren *et al.,* 2001).

The importance of solubility testing is highlighted because the amount of leachable residual monomers is closely linked to the degree of conversion of the material. Light curing, a crucial process for monomer conversion, must be optimized to reduce the amount of leachable monomers and ensure the biocompatibility of the material (Leprince *et al.,* 2013). In addition, the molecular weight of the monomers also influences solubility, as molecules of lower molecular weight dissociate more easily, releasing residual monomers into the oral cavity (Ferracane, 1994). This knowledge is vital for the development of new dental materials that are both durable and safe for patients.

## SUMMARY TABLE

Table 1: Schematic summary of the physicochemical and mechanical tests



**Mechanical testing in the evaluation of composite resin**

**Physicochemical test in the evaluation of composite resin**

FLEXIBLE STRENGTH

- Determine the ability to withstand deformations before failure;
- Simulates clinical conditions of masticatory stress;
- Three-point is the most common and widely used test for composite resins.

KNOOP MICROHARDNESS

- Assess surface hardness quantitatively;
- It is an indirect indicator of the degree of conversion of monomers into polymers;
- In composite resin: Knoop (ISO 4049).

SORPTION AND SOLUBILITY

- Evaluate the interaction of the material with fluids;
- Sorption: the material's ability to absorb liquids from the environment;
- Solubility: the amount of substances that the material releases into the liquid medium;
- Clinical importance: durability, aesthetics, adhesion and longevity.

Source: Authors, 2024.

## CONCLUSION

Physicochemical and mechanical tests play a key role in evaluating the quality and clinical performance of dental materials, such as composite resin. Of which, the flexural strength test can provide information on the material's ability to withstand masticatory forces, the microhardness test in the evaluation of the material's surface resistance to wear, and the sorption and solubility tests in identifying the material's interaction with the oral medium and its degradation potential. Minimum values expected by the ISO standardization of these tests are requirements in the development of more durable and reliable materials, as well as to assist in the choice of products that meet clinical demands, contributing significantly to the success and longevity of dental treatments.

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